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## TRANSITION FROM PLASTIC TO BRITTLE BEHAVIOR IN THIN TUNGSTEN WIRES

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### Introduction

Metals with a cubic body-centered crystal structure have the property to show the turn from the brittle to the ductile behavior during a deformation test, within a relatively narrow temperature range. This effect known as cold shortness was so far demonstrated in iron and steel, chromium, niobium, molybdenum and tungsten [1-4].

The notch-test (DIN 50515) is mostly used to establish cold shortness. Due to relatively large dimensions of the test samples, this method is not suitable for testing wires. The results of the tensile tests which were used for the wire tests [3-5] are likewise not applicable to the torsion- and bending-stresses which occur frequently in practice.

On the contrary, the method of examination of tungsten wires 20 to 500  $\mu$  in diameter described herein furnishes a direct disclosure as to the deformability of the thin wires and allows to establish with satisfactory accuracy the transition from plastic to brittle behavior.

### Method of Examination

In order to imitate the stresses during the processing of the tungsten wires, coils are made from the wire which is to be tested. For this reason the tungsten wire is coiled, with a coiling stress of about 10% of the tensile strength, on a molybdenum core wire. The rate of the bending stress is defined by the core factor (radius of the core to radius of the tungsten wire).

The coil is annealed in a forming gas (20%  $H_2$ , 80%  $N_2$ ) at sufficient high temperature in order to achieve the spacing according to the spacing of the adjoining core.

The test is conducted by placing the coils between two steel plates with a certain temperature. The plates are driven one against the other by a hammer stroke or by a striker. In this way the inserted coil is deformed as much as possible. The coil which conducts itself as completely ductile, shows on all outward places the largest possible bending deformation (Fig. 1).

The brittle wire material shows up at the places where the coil is broken. The number of breaking points and the length of the broken pieces is conclusive for the grade of the embrittlement.



Fig. 1. Plastic Deformation of a Tungsten Coil During the Stroke Test of the Coil.

#### Testing Apparatus

The testing apparatus consists of a bottom pan and a lid made of heat resistant steel. The coil placed between the bottom and the lid is compressed by a hammer stroke (stroke-cup method).

The advantage of this method is that the temperature of the coil at breaking point can be changed with sufficient accuracy with the variation of the stroke-cup temperature in the range between about  $-200$  and  $+700^{\circ}C$ . The stroke-cup temperature can be maintained constant and can be increased.

The speed of deformation is here only of little influence on the result of the measurements. However, in order to secure unequivocal conditions it is expedient to use a defined stroke impulse. In the described tests a weight of 170 gram felt from a height of 25 cm on the 140-gram stroke-cup lid.

For the sake of the constancy of the speed of deformation a device was designed in which the steel plates are drawn together by the action of a striker\*. However, due to heavy heat discharge, it is possible to work comfortably with this device only within a range of  $-50^{\circ}C$  to  $300^{\circ}C$ .

\*(Fig 2)

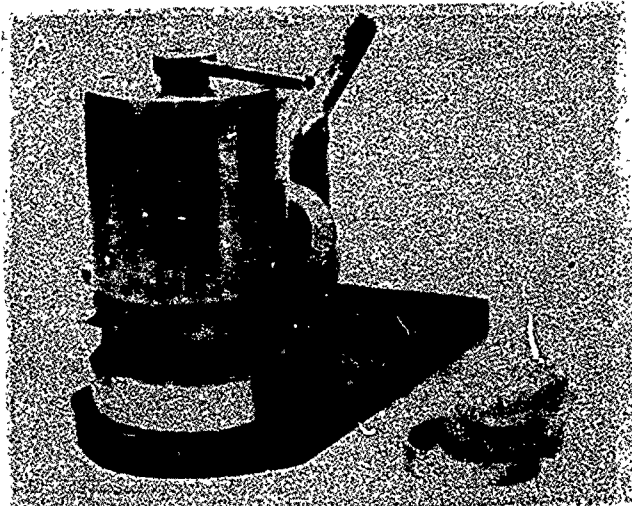


Fig. 2. Striker Apparatus for Coil Stroke Test.

#### Transition From Plastic to Ductile Behavior

In the coil-stroke test of the tungsten wires the transition temperature from brittle to ductile behavior always can be determined. At lower temperatures one can see only very small broken pieces (brittle fracture) whereas at higher temperatures the coil continues to remain in one piece (plastic zone). The intermediate range of temperature represents the transition zone from brittle to plastic behavior (Fig. 3). The experiments show that this range of temperatures for the tungsten wires is maximum about  $40^{\circ}\text{C}$ . Since the measuring accuracy is about  $20^{\circ}\text{C}$ , it is sufficient to give only one value for the transition temperature.

In our tests the temperature is always given at which about 10% of the bending places show fracture. This temperature will be henceforth defined as the embrittlement temperature  $T_e$ . This value of  $T_e$  marks the temperature at which the brittleness starts. At room temperature the tungsten wire is the more susceptible to breaking due to brittleness, the higher is  $T_e$ .

#### Results

The lowest obtained temperature  $T_e$  was at  $-100^{\circ}\text{C}$ . Some coils which were embrittled by annealing or through the influence of a contaminant had a  $T_e$  of more than  $700^{\circ}\text{C}$ . The position of  $T_e$  depends also considerably on the structure and on the treatment of the tungsten wire.



Fig. 3. Transition From Plastic to Ductile Behavior During the Stroke Tests of a Tungsten Coil.

1 -- Temperature of the stroke.

For drawn tungsten wires with fibrous structure, which are not annealed after coiling,  $T_e$  is always below  $0^\circ\text{C}$  and averages  $-50^\circ\text{C}$ . The influence of the wire diameter within a range of  $20\text{-}100\ \mu$  is not discernible. Therefore, such wires can be bent and twisted at room temperature.

Wires made of various commercial tungsten wire materials show characteristic differences after annealing.

1. Purer tungsten wire manufactured according to the Coolidge process (KD)\* loses already at  $1200^\circ\text{C}$  its fibrous texture and recrystallizes polygonally.

2. By adding alkali-silicates (NS) and also aluminum oxide (BSD) it is possible to achieve longitudinal

\*The marking given henceforth in parentheses will denote the commercial brands of the OSRAM Corporation.

crystallization (secondary recrystallization) at temperatures of 1800-2000°C. Thus are formed the longitudinal crystals which in the case of wires below 200  $\mu$  fill all the section of the wire. Below this temperature of secondary recrystallization, there is only a slow widening of the fibers (primary recrystallization).

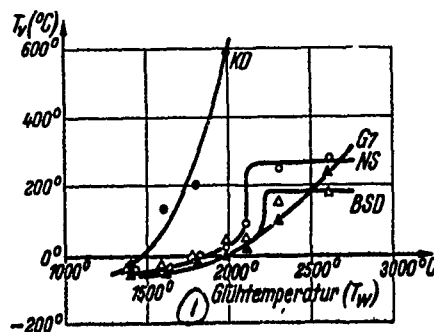


Fig. 4. Relationship Between  $T_g$  for Various Tungsten Wire Materials and the Incandescent Temperature.

1 -- Incandescent temperature ( $T_w$ ).

3. By adding thorium oxide (G7 and G18), secondary recrystallization is moved up to the highest incandescent temperatures.

The characteristic changes of the structure during the incandescence of tungsten wires are reflected again in the behavior of  $T_g$ .

1. For KD wires the beginning of the recrystallization is followed by a steep rise of  $T_g$  (Fig. 4). The emerged polygonal texture reaches mostly a  $T_g$  value of above 700°C.

2. For NS and BSD wires  $T_g$  rises only slightly in the zone of primary recrystallization. In many cases  $T_g$  remains in this zone below room temperature. At the temperature of secondary recrystallization the  $T_g$  diagram shows a sharp corner. For recrystallized coils of BSD wire  $T_g$  reaches generally lower values than for NS wire.

3. The thoriated wires show no sharp corner in the  $T_g$  diagram, but only a gradual rise of  $T_g$  with the increase of the incandescent temperature, which corresponds to the slow enlargement of the grain.

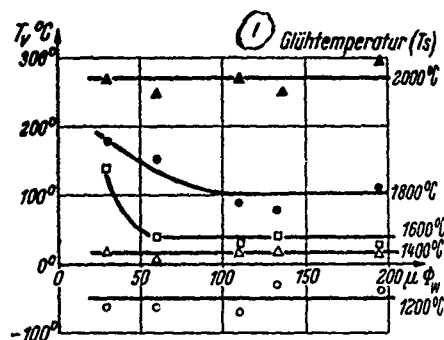


Fig. 5.  $T_e$  for Tungsten Coils of NS Material in Relation to the Diameter of the Wire and the Incandescent Temperature.

1 -- Incandescent temperature ( $T_s$ ).

Within a range 20-500  $\mu$  the diameter of the wire has only a limited effect on the position of  $T_e$ . Fig. 5 shows the measurements of  $T_e$  on wires of various diameters after incandescent treatment at various temperatures. The effect of the wire diameter is not discernible either at low incandescent temperatures or for recrystallized wires\*. The thin wires which we measured show in the zone of the middle temperatures (1600°C) slightly higher  $T_e$ . Presumably this effect can be traced back to the influence of contamination described below which is substantially more in thin wires.

#### Disturbances Due to Contaminants

The presence of various contaminants on the surface or in the ambient atmosphere embrittles the tungsten wire at incandescence.

Carbon as well as gases containing carbon form a hard and brittle film of carbide on the surface of the wire.

Other contaminants, particularly oxygen, as well as all metals of the iron (6-8) and platinum group, neutralize the effect of the additions to the tungsten wire and in a temperature range of 1100-1700°C lead to a very brittle small-crystalline or polygonal texture. The values of  $T_e$  of such tungsten wires which, due to the influence of contaminants, were prematurely recrystallized KD wires, very high, often above 700°C.

#### Conclusions

Our observations lead to the conclusion that the transition temperature from brittle to plastic behavior ( $T_e$ ) is

\* (2000°C)

determined mostly by the pattern of the grain boundaries in the tungsten wire.

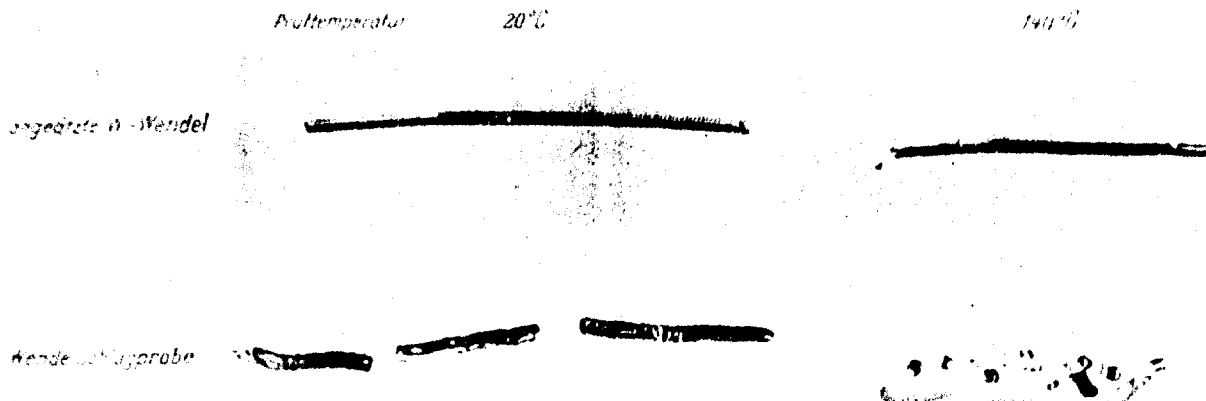


Fig. 6. Inter-Crystalline and Trans-Crystalline Fracture in Long Crystalline Tungsten Wire at Various Test Temperatures.

1 -- Test temperature; 2 -- Etched tungsten coils; 3 -- Coil-stroke-test.

On recrystallized wires with polygonal structure fractures appear only along the boundaries of the grain [1, 9]. The  $T_e$  of such a texture lies above  $700^\circ\text{C}$ .

Long-crystalline wires with distinct threshold of secondary recrystallization have usually highly coherent crystal boundaries. However, in such secondary recrystallized wires the fractures occur generally at the boundaries of the grains, but the whole surface of the fracture cannot run intercrystalline. Some transcrystalline proportion is to be expected. The values  $T_e$  lie here approximately between  $200$  and  $400^\circ\text{C}$ . Long-crystalline material tends distinctly towards lower values of  $T_e$ .

In wires with fiber structure the proportion of fractures along the grain boundaries is very small. It appears basically only in a transcrystalline fracture.  $T_e$  lies below room temperature.

The different position of the embrittlement temperature  $T_e$  for distinct structures of tungsten wires is also to be led back to the corresponding different values of  $T_e$  for fractures along grain boundaries and transcrystalline fractures.



The distinct position of  $T_e$  at the transcrystalline fracture and the fracture along the grain boundaries can be easily proved in the coils with particular long crystals which are stretched over many windings. The crystal lengths of such coils are made visible in Fig. 6 through surface etching. During the test at room temperature, the length of the individual fracture pieces corresponds exactly to the length of the crystals. Thus fractures appear only on the grain boundaries. At very low temperatures ( $-140^\circ\text{C}$ ) the individual crystals crush into many pieces. Thus also the monocrystal shows a brittle transcrystalline fracture at very low temperatures.

#### Summary

A method for testing the behavior of thin tungsten wires at bending deformation is described.

All tungsten wires show, within a narrow temperature range, the transition from brittle to ductile behavior. For wires with a fiber texture this transition temperature  $T_e$  is below  $0^\circ\text{C}$ ; for recrystallized wires, at  $150-400^\circ\text{C}$ . Long-crystalline wires have a lower  $T_e$  than the short-crystalline ones.

The position  $T_e$  is determined by the form of the fracture (transcrystalline resp. intercrystalline).

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